

Petroleum

## Acid number of insulating oil

Acid-base titration (non aqueous) by  
Automatic Potentiometric Titrator

<b>Standard</b>	<b>JIS C 2101</b>	<b>BS 14 8</b>
	<b>JPI-5S-57-99</b>	
	<b>ASTM D 974</b>	

### 1. Abstract

To maintain the acid number within specified level is the key of quality control of the insulating oil. Acid number is expressed by potassium hydroxide in mg required to neutralize free fatty acid in 1g of oil.

Measurement of acid number of insulating oil is specified in JIS-C2101 Test Method for Electrical Insulating Oil and JPI-5S-57-99 Neutralization Test Method for Electrical Insulating Oil – Total Acid Number Indicator Photometric Titration.

The oil sample is titrated with 0.05mol/L potassium hydroxide ethanol as reagent and with toluene ethanol (3+2) as solvent and with alkali blue 6B ethanol, and the endpoint is detected on titration curve. The acid number is calculated from the titration volume of reagent reaching the endpoint.

### 2. Reference

- 1) JIS-C2101-1999 Test Method for Electrical Insulating Oil
- 2) JPI-5S-57-99 Japan Petroleum Institute - Neutralization Test Method for Electrical Insulating Oil – Total Acid Number Indicator Photometric Titration
- 3) ASTM D974-06 Standard Test Method for Acid and Base Number by Color-Indicator Titration
- 4) BS148-1998 Specification for unused and reclaimed mineral insulating oils for transformers and switchgear

### 3. Cautions in measurement

- 1) The indicator of alkali blue 6B is hard to dissolve in ethanol, and needs to be stirred more than 2 hours before titration.
- 2) The indicator is liable to change, and recommended to use within 2 days after prepared.
- 3) Be aware the accuracy of measurement depends on the correct amount of indicator when added during the process of titration.

### 4. Post-measurement care

No special note

## 5. Test equipment

Main unit: Automatic potentiometric titrator (Photometric preamplifier: PTA—)  
Detector : Photometric electrode 12-001-100-48 ( $\lambda = 650\text{nm}$ )

## 6. Reagent

Reagent : 0.05mol/L potassium hydroxide·ethanol solution (  $f=0.6823$  )  
Solvent : Toluene + ethanol (3+2) mixture  
Indicator : Alkali blue 6B ethanol solution

## 7. Test procedure

—Preparation—

- 1) Obtain the factor of reagent using amide sulfuric acid.
- 2) Dissolve 0.1~0.2g alkali blue 6B in 100mL ethanol, and filtrate as indicator.
- 3) Calibrate transmissivity with 100mL solvent added with 3mL indicator and 2mL reagent as 100%T, and with 100mL solvent added with 3mL indicator as 0%T respectively.

—Measurement—

- 1) Transfer 20g sample to a 200mL beaker, and weigh it on a balance.
- 2) Add 100mL solvent and 3mL indicator.
- 3) Titrate with 0.05mol/L potassium hydroxide ethanol.
- 4) Perform a blank test in advance to obtain blank level.

## 8. Formula

Acid number ( mgKOH / g ) = ( EPl - BLl ) × TF × Cl × Kl / S

EPl : Titration volume ( mL )

BLl : Blank level ( 0.2999mL )

TF : Reagent factor ( 0.6823 )

Cl : Concentration conversion coefficient ( 56.1 g/mL )

(equivalent to potassium hydroxide in 1mol/L potassium hydroxide ethanol)

Kl : Unit conversion coefficient ( 0.05 )

S : Sample size ( g )

## 9. Example of measurement

— Ambient condition —

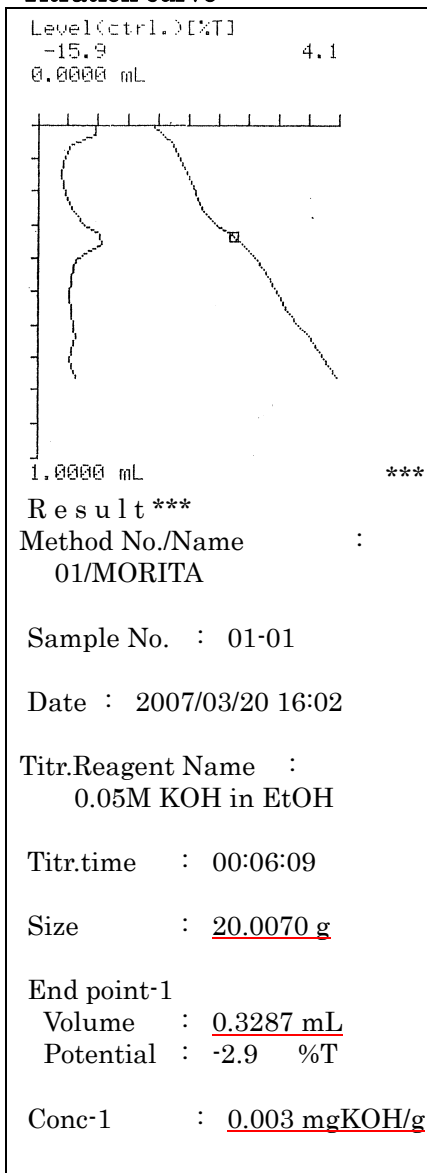
Room temperature : 20.0 □	Humidity : 30 %	Weather : Fair
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The following parameters and titration curve were obtained by AT-610 titration system:

### -Titration parameter-

Model : AT-610	
Method No. : 01	
Titr.mode : Intermit	
Titr.form : EP Stop	
[Titration parameter]	[Calculation parameter]
Burette No. : 1	Calc. Type : Sample
Max volume : 1.50 mL	CO1 : On
Channel,Unit	Unit : mgKOH/g
Ctrl. : Ch3,%T	Formula :
Ref. : Off	(EP1-BL1)*TF*K1*C1/S
pH polarity :	EP position : EP1
Standard	Decimal : 3
Direction : Auto	Fraction : Half adjust
Wait time : 0 s	Evaluation : Off
Dose mode : None	
[Control parameter]	Active constant
Number of EP : 1	C1 : 56.1
End sense : Auto	K1 : 0.05
Gain : 1	Temp.comp. : off
Data sampling : Set	
Data sampling : Set	
Data samp.pot. : 4.0 mV	
Data samp.vol. : 0.05 mL	
Ctl.speed mode : Set	
Cut-off time : 10 s	
Unit volume : 0.025mL	
Disp speed : 1 s/mL	
Other control : Std.	
Stir. Speed : 3	

### -Titration curve-



### Meaning of printout data:

#### «Titration parameter»

Burette No.: the burette used in titration / Max Volume: of titration  
Channel,Unit: the detector used in potential detection and the unit of potential selected  
pH polarity: direction of pH potential / Direction.: of titration / Wait Time: before titration starts  
Dose mode: for fixed dose

#### «Control parameter»

Number of EP: number of endpoints detected / End sense: direction of EP sense / Gain: of detection signal  
Data sampling: conditions / Ctl.speed mode: control speed mode  
Other control: other controls / Stir. Speed: of stirrer during titration

#### «Calculation parameter»

Calc.Type: calculation type / CO1: concentration formula 1 /Unit: unit of calculation results  
EP1: titration volume to reach EP1 / BL1: blank level / TF: factor of reagent  
C1(mg/mL): concentration conversion coefficient / K1: unit conversion coefficient  
S: sample size / EP position: for calculation / Decimal: number of digits after decimal point in calculation  
Fraction: rounding fraction / Evaluation: of calculation results

—Measurement results—

n	Sample (g)	Titrated (mL)	Acid number (mgKOH/g)	Statistics	
				Mean	SD
1	<u>20.0070</u>	<u>0.3287</u>	<u>0.003</u>	0.003 mg/g	0.0006 mg/g
2	20.0090	0.3228	0.002		
3	20.0096	0.3313	0.003		20.0 %

\* The above results were obtained from 3 tests of the same sample.

\* Red underline shows the data from page 3/4.

## 10. Summary

Acid number is expressed by potassium hydroxide in mg required to neutralize free fatty acid in 1g of oil. To maintain the acid number within specified level is the key of quality control of the insulating oil.

The measurement results of the sample this time show a good repeatability.

The automatic potentiometric titration system provides stable and reliable measurements. The acid number of insulating oil is measured by any of the models shown below.

### 【AT-610】



**Awarded Product of Supreme Technology from Kyoto City**

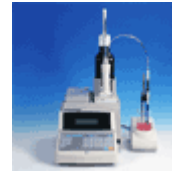
- Easy key entry by touch panel of large color LCD (8-inch wide)
- Simultaneous titration in parallel
- Both potentiometric and Karl Fischer moisture titration (coulometric • volumetric) can be performed at a time.

### 【AT-510】



- Compact and cost performance model
- PC card expands data memory for convenience and versatility.

### 【AT-500N-1】



- Low cost and high performance
- Easy view with back light LCD
- GLP/GMP conformed model

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